# Improved Manostat and Manometer

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ALTHOUGH many pressure controllers suitable for use in vacuum distillations have been reported (*5*), it is believed that the manostat described here is better adapted to precision vacuum fractionation than those previously described. A novel feature is the use of a completely enclosed magnet-operated screw, which permits rapid and precise adjustment to any pressure over a wide operating range. The manostat is substantially independent of small variations in room temperature or pump capacity. Moreover, it is small and light in weight, and may be clamped conveniently to the usual laboratory rack or ring stand. Its construction requires the services of a skillful glassblower. A manufacturer of scientific apparatus is now developing a model for commercial production. Because deviations from the controlled pressure depend not only on the pressure-sensitive element, but also on the rest of the system, Figure 1 gives a semischematic representation of the arrangement used to obtain the degree of constancy reported here.

The modified 40 × 170 mm. borosilicate glass test tube, 13, has an 8-mm. sidearm, 10, and a 14/38 ground-glass neck, 9. It holds approximately 40 ml. of mercury. The amount is not critical, but should be enough to fill the central tube completely, in case tube 13 is allowed to come to atmospheric pressure.

The 8-mm. central tube, 12 (soft glass ground to size 14/38), extends to within a few millimeters of the bottom of tube 13 and terminates in a 0.5-mm. hole, 15. The upper portion, 12 × 160 mm., contains two glass tabs, which hold stainless steel bracket 7; this bears two extensions, 25 and 8; 25 serves as a bearing for the stainless steel screw, 4 (50 threads per inch), and 8 serves as pivot for the screw. The upper end of the screw terminates in a soft iron vane, 3. On the screw is threaded stainless steel nut 5, of such size that when the screw is turned the nut is prevented from turning with it by the bracket, and so must advance or retreat along the screw. Attached to nut 5 is a stiff tungsten wire lead, 11, which is connected electrically to the outside of the central tube at the top through the nut, screw, bracket, and platinum wire 27, which is sealed through the tube.

The central tube also contains the platinum lead, 14, which is in contact with the mercury at all times and is held against the side by a thin glass tube tacked to the wall. This lead is also sealed through at the top, thus making the second of terminals 1, 1

The seals at the top are protected by thermoplastic cement, which also holds in place the metal guide, 26, on which rests the annular magnet, 2. As the magnet is turned, it turns vane 3 and thus screw 4, moving the tip of the tungsten electrode axially within the tube.

The terminals are connected to an electronic relay, 28, which operates solenoid 22 (1000 ohms). Use of an electronic relay minimizes the probability of sparking at the contact between

electrode 11 and the mercury. A spark is undesirable, especially where flammable materials or explosive vapors may be present in the confined space.

The solenoid is connected to the relay, so that when the manostat circuit is broken, plunger 23 (faced off with a soft rubber pad, 24) is drawn up, thus opening orifice 16.

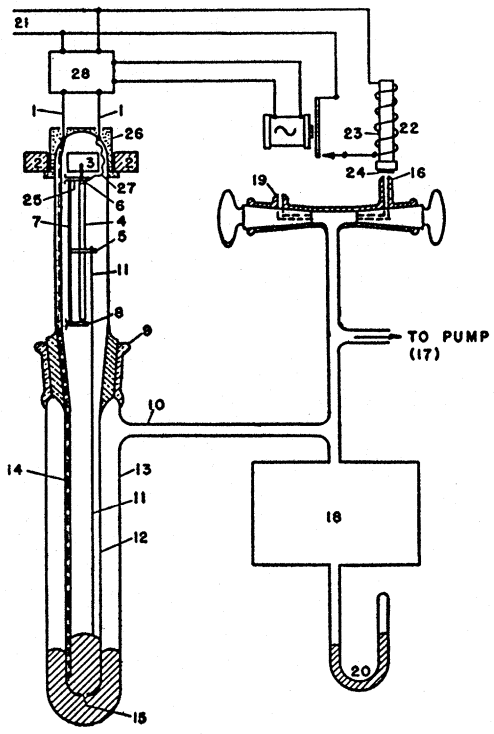


**Figure 1. Diagram of Manostat**

The flow of air into the system and pump is controlled by a special stopcock, with independent adjustment of a continuous leak through orifice 19, and adjustment of the intermittent leak through orifice 16. The plug of each stopcock is carefully grooved to permit throttling of gas flow. Orifice 16 is a 1-mm. capillary ground to a conical shape and then ground flat on the sealing surface.

To place in operation, side arm 10 is connected with pressure tubing to a good pump, and the manostat is tilted to expose hole 15 and thoroughly evacuated. The manostat is then restored to the upright position; the side arm is clamped off, detached from the pump, and attached to the system to be controlled, 18.

Pump 17 is started, and with orifice 16 closed by the stopcock, the system is pumped to a few millimeters below the desired pressure by adjustment of orifice 19. At the lower pressures, orifice 19 is completely closed. The pinchclamp on the manostat is next opened, and the position of electrode 11 is adjusted to open the circuit and lift the plunger. The stopcock on the right is then opened slightly to permit the pressure to rise slowly. As soon as the mercury touches electrode 11, the relay permits the plunger to fall on orifice 16. The pressure begins to fall and the mercury is withdrawn from contact 11, whereupon the plunger rises, and the cycle repeats.

With proper adjustment of the stopcocks, operation will occur with a slight "breathing" motion of the mercury meniscus. If the equilibrium pressure in the system, measured, for example, by manometer 20, is not that desired, it is quickly brought to the correct point by turning the magnet (one complete turn of the screw advances the nut 0.5 mm.). Adjustments can be made to a more precise degree than can be read on the usual U-tube manometer.

The manostat prepared in this way is also an absolute manometer. For some purposes, measurement by this manometer may be sufficient, but it is usually desirable to connect a second manometer directly to the system. When the manostat is to be used as a manometer, a millimeter scale is fastened to the side of the tube, or may be etched directly on tube 13.

If it is necessary to control pressures higher than about 100 mm., the manostat is tilted on its side, and air is admitted to the central tube while the system is near the desired pressure. If air at 100 mm. is admitted, the new operating range is 100 to 220 mm.; if air at 220 mm. is admitted, the range is 220 to 375. The range 3 to 760 mm. can be covered in five steps. Explosive vapors should be excluded from the central tube, even though the use of an electronic relay minimizes the probability of sparking.

It is preferable to connect the manostat and leak by a T-type connection near the pump. With an ordinary distilling column no surge chamber between the system and manostat connection is necessary, but if the controlled system has a small volume or if the pressure is high (150 mm.), a surge chamber will help smooth out the pressure variations caused by the controlling action.

It is unnecessary to clamp off the manostat when fractions are changed; admission of air into tube 13, as in evacuating a fresh receiver, causes no permanent change in the system; controlling action resumes when the mercury level reaches the end of the electrode.

In some cases of this kind, however, cohesion of the mercury and glass results in a temporary control point 0.1 to 0.2 mm. away from the initial pressure, to which the controller readjusts rather slowly. Hence, it may be advisable to tap the manostat or make an adjustment.

The manostat retains all the advantages of an adjustable electrode (*1*), but avoids the uncertainties and difficulties of leakproof packing (*2*); moreover, the controlling action in the range 3 to 100 mm. occurs in a vacuum, where fouling of the mercury is less likely to occur.

Because the manostat and relay, in effect, constitute a switch, modifications in the leak system could be made to meet various requirements. The relay could be used to switch a pump (*2*, *7*), operate a breather valve between system and pump (*6*), or bleed inert gas to a system by means of a breather valve (*4*).

The authors have used the manostat to control the distillation pressures for a series of boiling point measurements (*3*), and to check reported boiling point measurements. The lowest pressure at which it was operated was 3.0 mm. Orifice 19 was closed off entirely, and after preliminary adjustment the pressure remained constant for several hours, without any visible variation in pressure apparent on a Dubrovin gage, which is calibrated in 0.2-mm. intervals.

At 144.0 mm., the pressure remained constant within ±0.1 mm. for 5 hours. The highest pressure at which the manostat was operated was 523 mm.

At the highest pressure, the capacity of the pump exceeded that of the leaks, and it was necessary to throttle the pump with a pinchclamp. The test was run for 3 hours; the pressure remained at 523 ± 0.2 mm.


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## LITERATURE CITED


(1) Cox, H. L., Ind. Eng. Chem., Anal. Ed., 1, 7–8 (1929).
(2) Ellis, L. M., *Ibid.*, 4, 318–19 (1932).
(3) Fein, M. L., and Fisher, C. H., *J. Org. Chem.*, 13, 749 (1948).
(4) Jacobs, G. W., Ind. Eng. Chem., Anal. Ed., 7, 70 (1935).
(5) Morton, A. A., "Laboratory Technique in Organic Chemistry," pp. 105–9, New York, McGraw-Hill Book Co., 1938.
(6) Palkin, S., and Nelson, O. A., Ind. Eng. Chem., Anal. Ed., 6, 386 (1934).
(7) Willingham, C. B., Taylor, W. J., Pignocco, J. M., and Rossini, F. D., *J. Research Natl. Bur. Standards*, 35, 219 (1945).